Journal of
Environmental
Nanotechnology

# Impact of Reducing Agent on the Synthesis of Nickel Oxide Nanoparticles by Chemical Precipitation Method

V. Kayathri, K. Kousalya, A. Mafeena, M. Monisha, S. Naga Nandhini,
J. Balavijayalakshmi*
Department of Physics, PSGR Krishnammal College for Women, Coimbatore, TN, India
Received: 13.01.2021 Accepted: 03.03.2021
*balavijayalakshmiroopa@gmail.com

## ABSTRACT

This present work reports the synthesis ad characterization of Nickel oxide nanoparticles by chemical precipitation method from nickel chloride by using two different reducing agents, such as ammonia ($NH_3$) and sodium hydroxide (NaOH). The prepared nanoparticles are characterized by X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Field Emission-Scanning Electron Microscopy (FE-SEM) and Energy Dispersive X-ray analysis (EDAX) techniques. XRD analysis showed that the prepared nanoparticles are crystalline in nature, and the average crystallite size for NiO nanoparticles with NaOH and NiO nanoparticles with $NH_3$ is found to be 25.3 nm and 27.54 nm, respectively. FE-SEM analysis revealed that the prepared nanoparticles are spherical in shape with agglomeration. The presence of functional groups of the prepared nanoparticles is confirmed by using FT-IR analysis. The band at 450 $cm^{-1}$ corresponds to the stretching vibration on Ni-OH, which confirms the presence of NiO, and the elemental analysis confirms the presence of nickel and oxygen elements in the prepared nickel oxide nanoparticles without any Impurities. The synthesized nickel oxide nanoparticles may be used in micro-supercapacitors, electrochromic coatings, and chemical sensing devices.

**Keywords:** Chemical precipitation; Nickel oxide; Reducing agent.

## 1. INTRODUCTION

Nickel oxide (NiO) is the most widely investigated transition metal oxide. It is a NaCl-type antiferromagnetic oxide semiconductor. It offers promising candidature for many applications such as solar thermal absorber, the catalyst for $O_2$ evolution, photoelectrolysis and electrochromic device. Nickel oxide is also a well-studied material as the positive electrode in batteries (Ibraheem *et al.* 2019; Lingaraju *et al.* 2019). Pure stoichiometric NiO crystals are perfect insulators. Several efforts have been made to explain the insulating behaviour of NiO. Appreciable conductivity can be achieved in NiO by creating Ni vacancies or substituting Li for Ni at Ni sites. The most attractive features of NiO are excellent durability and electrochemical stability, low material cost, promising ion storage material in terms of cyclic stability, large span optical density and possibility of manufacturing by a variety of techniques. Nickel oxide has a good electrochemical property; low toxicity and low cost are particular interest which makes them suitable for many applications (Patil *et al.* 2002). In this present work, nickel oxide nanoparticles are synthesized by chemical precipitation method from Nickel chloride by using two different reducing agents such as ammonia ($NH_3$) and sodium hydroxide (NaOH) (Nassar *et al.* 2017).

The chosen metal oxide nanoparticles, like nickel oxide, is an important transition metal oxide with a cubic lattice structure and of p-type semiconducting material with a bandgap of 4.0 eV. The purpose of the study is to explore the feasibility to synthesize NiO nanoparticles by chemical precipitation method from Nickel chloride by using two different reducing agents such as ammonia ($NH_3$) and sodium hydroxide (NaOH). The results obtained by characterization techniques that involve X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Field Emission-Scanning Electron Microscopy (FE-SEM) and Energy Dispersive X-ray Analysis (EDAX) is reported.

## 2. MATERIAL & EXPERIMENTS

### 2.1 Materials

Nickel chloride, sodium Hydroxide (NaOH) and Ammonia ($NH_3$) are purchased from Sigma Aldrich and used without further purification.

### 2.2 Synthesis of Nickel Oxide Nanoparticles

NiO nanoparticles are prepared by the chemical precipitation method. 8.5g of nickel chloride is dissolved in 80 ml of distilled water. The solution of light green

colour is obtained, which is sonicated for an hour, and the particles are agitated using ultrasonic vibrations (Singh *et al.* 2016). The sonicated solution is stirred into which NaOH is added drop by drop as a reducing agent to maintain a pH value of about 8. The process of stirring is carried with 450 rpm at 45 °C for about 4 hrs. Then the solution is kept undisturbed for 24 hrs to obtain a green precipitate which settles in the bottom of the beaker. The filtrate is centrifuged several times at 2000 rpm with each cycle of duration 10 minutes using centrifugal force to separate fluid of different densities. After the process of centrifuge, the resulting white precipitate is cleaned. The white precipitate is dried at 60 °C for about 4 hrs to allow ionic movements by heating the particles at elevated temperature. The precipitate is calcinated under 450 °C for about 4 hrs. The obtained particles are grained into fine particles of powder form using mortar and pestle. The same procedure is repeated to synthesize Nickel Oxide nanoparticles using ammonia as a reducing agent (T. Sone *et al.* 2016; Shanmugapriya *et al.* 2019).

### 2.3 Characterization Techniques

The prepared NiO nanoparticles' crystalline structure is examined using an XPERT-3 PANalytical Diffractometer. Shimadzu IR affinity-1 is used to record FT-IR spectra for the presence of nanoparticles. The morphology and microstructure of the sample are studied using field emission scanning electron microscopy (FESEM) using ZEISS-SIGMA. The elemental composition is tested using Energy Dispersive X-ray Spectroscopy (EDX).

## 3. RESULTS & DISCUSSIONS

### 3.1 XRD Analysis

The X-ray diffraction patterns of prepared NiO nanoparticles using ammonia ($NH_3$) and sodium hydroxide (NaOH) as a reducing agent is shown in Fig. 1 (a-b). Fig. 1(a) shows the diffraction peaks at 2θ values of 37.38°, 43.42°, 63.02°and75.53° corresponding to the (111), (200),(220) and (311) planes respectively and the Figure 1(b) shows the diffraction peaks at 2θ values of 38.2°, 43.4°, 63.0°and 75.3° corresponding to the (111), (200),(220) and (311) planes respectively and are well-matched with JCPDS card number 47-104, confirm the presence of NiO nanoparticles. The crystallite sizes for prepared nanoparticles of NiO($NH_3$) and NiO(NaOH) are found to be about 27.54 nm and 25.3nm, respectively, by using the Scherrer formula (Shanmugapriya *et al.* 2019).

### 3.2 FT-IR Spectral Analysis

The FT-IR spectrum of the prepared NiO nanoparticles with ammonia and sodium hydroxide as a reducing agent is shown in Fig. 2(a-b). It is observed from Fig. 2(a-b) that the broad absorption bands at 3440 $cm^{-1}$and 3460 $cm^{-1}$ are attributed to the O-H stretching vibrations of NiO ($NH_3$) and NiO (NaOH), respectively. The bands at 1647 $cm^{-1}$ and 1630 $cm^{-1}$corresponds to the H-O-H stretching vibration of NiO ($NH_3$) and NiO (NaOH), respectively (Bhavani *et al.* 2017). The bands at 683 $cm^{-1}$ and 702 $cm^{-1}$ represents the Ni-OH band stretching vibrations, where the oxygen combined with nickel-metal as a binary chemical compound of NiO ($NH_3$) and NiO (NaOH), respectively. The bands at 428 $cm^{-1}$ and 436 $cm^{-1}$ represents the Ni-O band stretching vibrations of NiO($NH_3$) and NiO(NaOH), respectively.

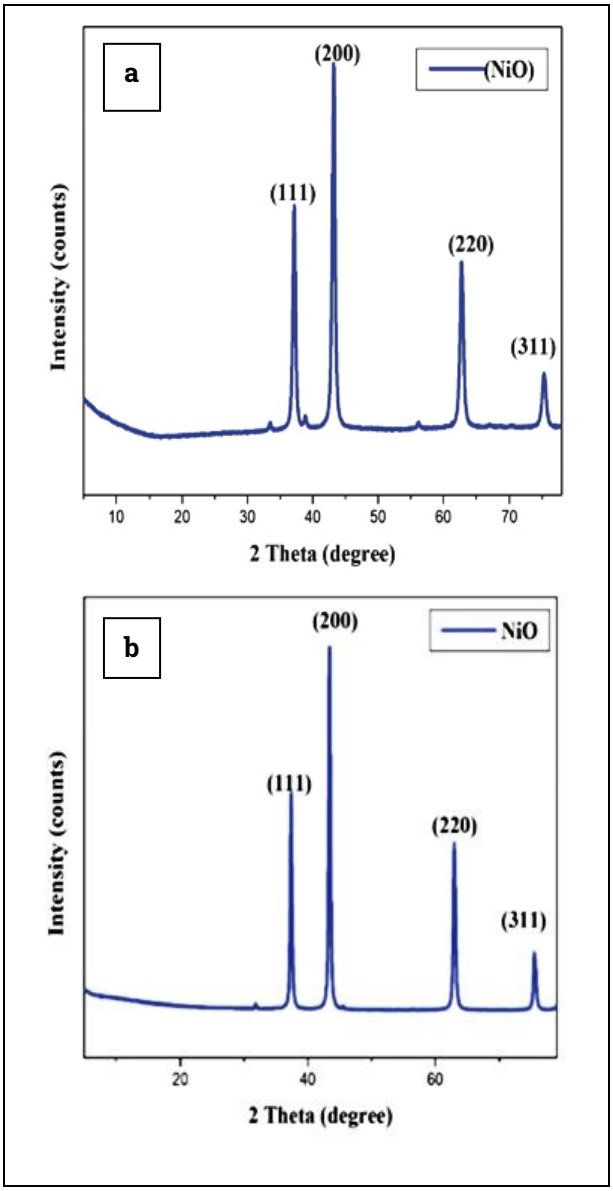


**Fig. 1: XRD spectra of NiO Nanoparticles using (a) Ammonia (b) Sodium Hydroxide as reducing agent**

Thus, the presence of NiO functional groups can be confirmed using FT-IR spectral analysis (Ali *et al.* 2018).

Fig. 3(a-b) shows the Field-Emission Scanning Electron Microscope images of NiO nanoparticles using $NH_3$ as reducing agent and Fig. 3 (c-d) shows FE-SEM images of NiO nanoparticles using NaOH as reducing agent. Fig. 3 (a-d) shows the morphological analysis of the prepared NiO nanoparticles and are in a spherical shape with agglomeration (Sun *et al.* 2017; Lassoued *et al.* 2018).

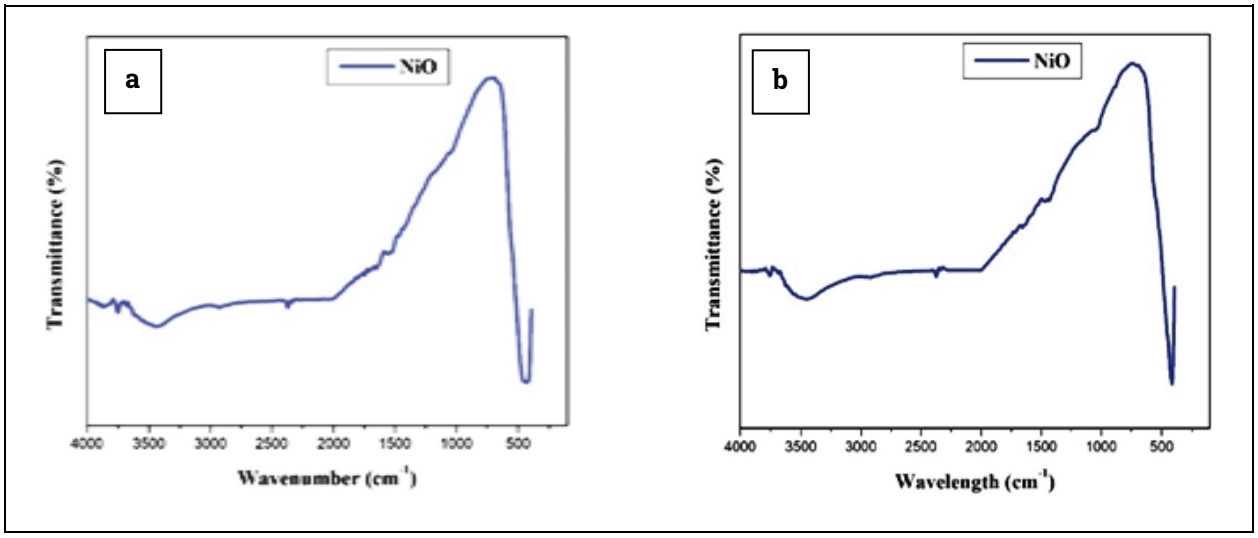


**Fig. 2: FT-IR spectra of NiO Nanoparticles using (a) Ammonia (b)Sodium Hydroxide as Reducing Agent**

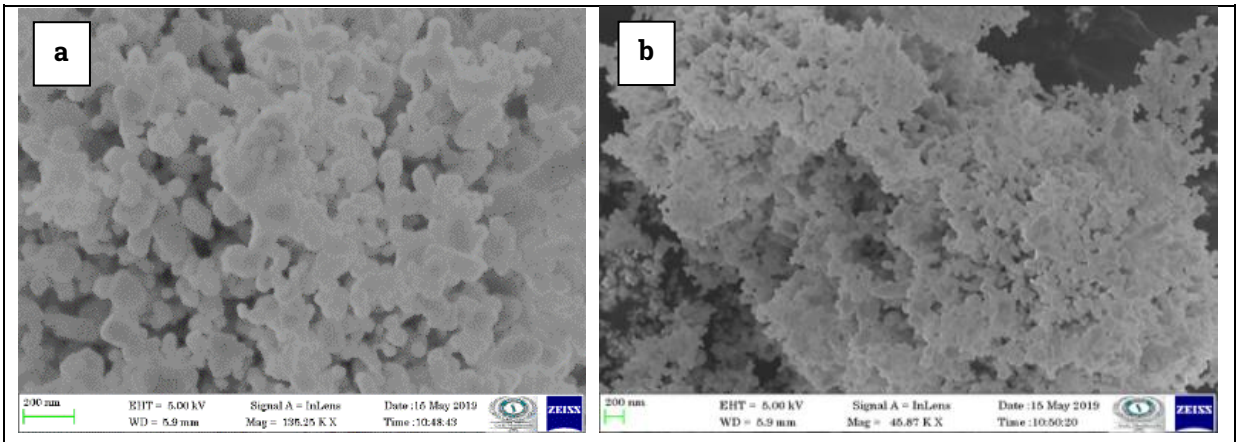


**Fig. 3(a-b): FE-SEM Images of NiO Nanoparticles UsingAmmonia as Reducing Agent**

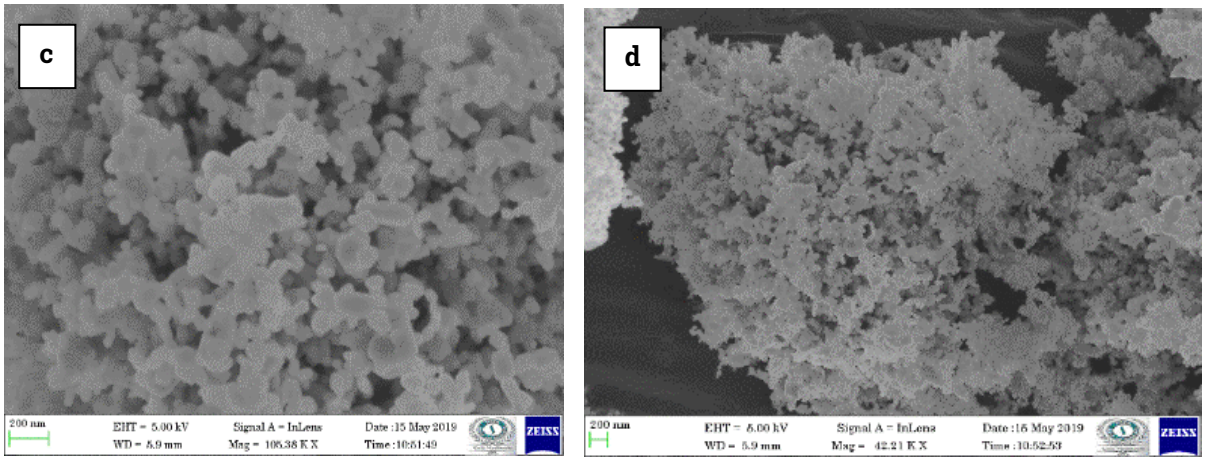


**Fig. 3(c-d): FE-SEM Images of NiO Nanoparticles using Sodium Hydroxide as Reducing Agent**

### 3.4 EDAX Analysis

Fig. 4 (a-b) shows the Energy Dispersive X-ray spectral analysis of NiO nanoparticles using $NH_3$ as reducing agent and NiO nanoparticles using NaOH as reducing agent respectively, and it is performed to know the elemental presence of NiO nanoparticles (Taghizadeh *et al.* 2016; Gandhi *et al.* 2016). Fig. 4 (a) shows that the element O K has an atomic weight of 52.15% and Ni K has an atomic weight of 47.85%, whereas Fig. 4 (b) shows that the element O K has an atomic weight of 76.73% and Ni K has the atomic weight of 23.27% and thereby confirms the elemental presence of nickel oxide nanoparticles without any impurities (Sharanabasava V.Ganachari, Ravishankar Bhat *et al.* 2012; Kavitha *et al.* 2016).

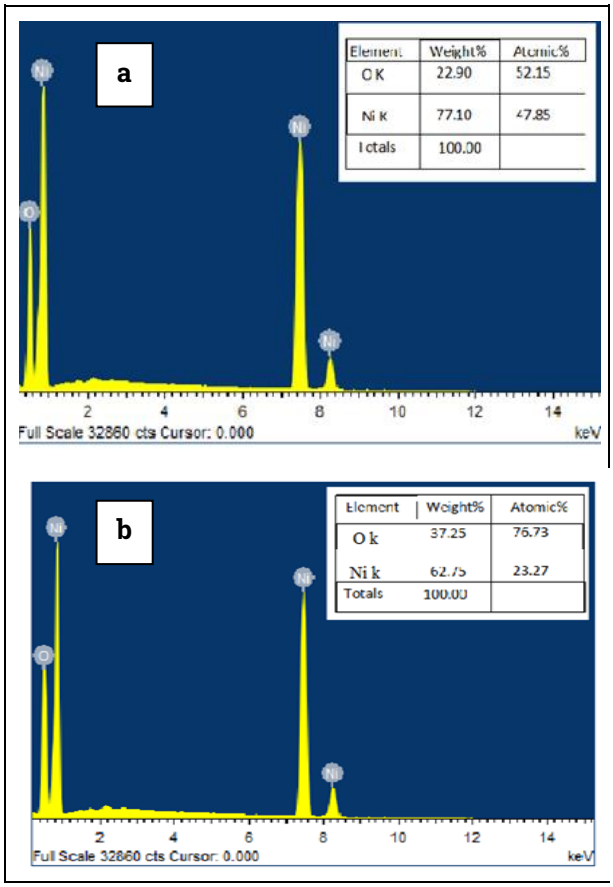


**Fig. 4: EDAX Analysis For NiO Nanoparticles using (a) Ammonia (b) Sodium Hydroxide as Reducing Agent**

## 4. CONCLUSION

Nickel Oxide nanoparticles have been successfully synthesized by chemical precipitation method from Nickel chloride by using two different reducing agents such as ammonia ($NH_3$) and sodium hydroxide (NaOH). The prepared nanoparticles are characterized by X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Field Emission-Scanning Electron Microscopy (FE-SEM) and Energy Dispersive X-ray Analysis (EDAX) techniques. XRD analysis showed that the prepared nanoparticles are crystalline in nature, and the average crystallite size for NiO nanoparticles with NaOH and NiO nanoparticles with $NH_3$ is found to be 25.3 nm and 27.54 nm, respectively. FE-SEM analysis revealed that the prepared nanoparticles are spherical in shape with agglomeration. The presence of functional groups of the prepared nanoparticles is confirmed by using FT-IR analysis. The band at 450 $cm^{-1}$ corresponds to the stretching vibration of Ni-OH, which confirms the presence of NiO, and the elemental analysis confirms the presence of nickel and oxygen elements in the prepared nickel oxide nanoparticles without any impurities. This present work concludes that the nickel oxide prepared using sodium hydroxide as a reducing agent is better than that of nickel oxide prepared using ammonia as reducing agents evident from XRD analysis due to the reduction in the crystallite size and also because of its environmentally friendliness and non-toxicity. The prepared Nickel oxide (NiO) can be employed in capacitor-inductor devices, tuned circuits, transparent heat mirrors, thermistor, batteries, micro-supercapacitors, electrochromic and chemical or temperature sensing devices.

## REFERENCES

Ali, A. A., El Fadaly, E. and Ahmed, I. S., Near-infrared reflecting blue inorganic nano-pigment based on cobalt aluminate spinel via combustion synthesis method, Dyes and Pigments, 158, 451-462(2018). https://dx.doi.org/10.1016/j.dyepig.2018.05.058

Bhavani, P., Rajababu, C., Arif, M., Reddy, I. V. S. and Reddy, N. R., Synthesis of high saturation magnetic iron oxide nanomaterials via low temperature hydrothermal method, *J. Magn. Magn. Mater.*, 426(C), 459-466(2017). https://dx.doi.org/10.1016/j.jmmm.2016.09.049

Ganachari, S. V., Bhat, R., Deshpande, R. and Venkataraman, A., Synthesis and characterization of nickel oxide nanoparticles by self-propagating low temperature combustion method, Recent Research in Science and Technology, 4(4), 50-53(2012).

Gandhi, A. C., Cheng, H. Y., Chang, Y. M. and Lin, J. G., Size confined magnetic phase in NiO nanoparticles, Mater. Res. Express., 3(3), 035017(2016). https://dx.doi.org/10.1088/2053-1591/3/3/035017

Ibraheem, F., Aziz, M. H., Fatima, M., Shaheen, F., Syed Mansoor Ali and Huang, Q., In vitro cytotoxicity, MMP and ROS activity of green synthesized nickel oxide nanoparticles using extract of Terminalia chebula against MCF-7 cells, *Mater. Lett.*, 234 (1), 129–133(2019). https://dx.doi.org/10.1016/j.matlet.2018.09.075

Kavitha, B., Nirmala, M. and Pavithra, A., Annealing effect on nickel oxide nanoparticles synthesized by sol-gel method, World Scientific News, 52, 118-129(2016).

Lassoued, A., Lassoued, M. S., Dkhil, B., Ammar, S., Gadri, A., Synthesis, photoluminescence and Magnetic properties of iron oxide ($\alpha$-$Fe_2O_3$) nanoparticles through precipitation or hydrothermal methods, Physica E: Low-dimensional Systems and Nanostructures, 101, 212-219(2018). https://dx.doi.org/10.1016/j.physe.2018.04.009

Lingaraju, K., Raja Naika, H., Nagabhushana, H. and Nagaraju, G., *Euphorbia heterophylla* (L.) mediated fabrication of ZnO NPs: Characterization and evaluation of antibacterial and anticancer properties, *Biocatal. Agri. Biotech.*, 18, 100894(2019). https://dx.doi.org/10.1016/j.bcab.2018.10.011

Nassar, M. Y., Aly, H. M., Abdelrahman, E. A., Moustafa, M. E., Synthesis, characterization and biological activity of some novel Schiff bases and their Co(II) and Ni(II)complexes: a new route for $Co_3O_4$ and NiO nanoparticles for photocatalytic degradation of methylene blue dye, *J. Mol. Struct.*, (1143), 462–471(2017). https://dx.doi.org/10.1016/j.molstruc.2017.04.118

Patil, P. S. and Kadam, L. D., Preparation and characterization of spray pyrolyzed nickel oxide (NiO) thin films, Applied Surface Science, 199(1-4), 211-221(2002). https://dx.doi.org/10.1016/S0169-4332(02)00839-5

Shanmugapriya, T. and Balavijayalakshmi, J., Preparation and characterization of nitrogen doped graphene oxide/nickel oxide nanocomposites for dye sensitized solar cell applications, Adv. Appl. Res., 11(1), 34-38(2019). https://dx.doi.org/10.5958/2349-2104.2019.00006.8

Singh, P., Kim, Y. J., Zhang, D., Yang, D. C., Biological synthesis of nanoparticles from plants and microorganisms, *Trends Biotechnol*., 34(7), 588–599(2016). https://dx.doi.org/10.1016/j.tibtech.2016.02.006

Sone, B. T., Fuku, X. G., Maaza, M., Physical and electrochemical properties of green synthesized bunsenite NiO nanoparticles via Callistemon viminalis extracts, *Int. J. Electrochem*. Sci., 11, 8204–8220(2016). https://dx.doi.org/10.20964/2016.10.17

Sun, D. L., Zhao, B. W., Liu, J. B., Wang, H. and Yan, H., Application of nickel oxide nanoparticles in electrochromic materials, *Ionics*, 23(6), 1509-1515(2017). https://dx.doi.org/10.1007/s11581-017-1974-4

Taghizadeh, F., The study of structural and magnetic properties of NiO nanoparticles, Optics and Photonics Journal, 6(8), 164-169(2016). https://dx.doi.org/10.4236/opj.2016.68B027